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(54) Title: METHOD FOR SURFACE TREATING ALUMINUM PRODUCTS		
(57) Abstract There is disclosed a method for surface treating aluminum products, especially vehicle wheels, to improve their brightness. The method comprises: (a) applying a chemical brightening composition to these products; (b) deoxidizing the surface of said wheel products in a nitric acid-based bath; (c) forming a porous oxide on that with an electrolytic bath containing phosphoric or phosphonic acid; and (d) applying, preferably spraying, a siloxane-based film to that porous oxide. In many instances, intermediate rinsing substeps are performed between these respective main steps.		

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METHOD FOR SURFACE TREATING ALUMINUM PRODUCTS

This application claims the benefit of U.S. Provisional Application Serial No. 60/098,320, filed on August 28, 1998, the disclosure of which is fully incorporated by reference herein.

5 This invention pertains to the field of methods for cleaning and surface treating aluminum products to improve their brightness. More particularly, the invention pertains to an improved, more efficient method for surface treating aluminum wheel products made by forging, casting and/or joining practices. Such wheels are suitable for automobiles, light trucks, heavy duty trucks and buses. This
10 invention may also be used to surface treat aerospace wheels and other aerospace components.

 Present surface treatments for bright aluminum products involve a plurality of separate steps including: cleaning, deoxidizing, chemical conversion and painting. Some of the foregoing process steps typically incorporate surface active
15 agents and/or corrosion inhibitors. The final painting step for many aluminum products is a polymeric clear coat applied in either a liquid or powder form. All these processes rely on the availability of bright aluminum surfaces for starting. Part of the overall success of these surface treatments hinges on minimizing initial brightness degradation during application of the known chemical treatments described
20 in more detail hereafter.

 Disadvantages with such prior art processes include:

1. They required a *starting* bright aluminum surface. The processes did not induce any brightness themselves.
2. The chemical treatment (i.e. cleaning, deoxidizing and chemical
25 conversion) and painting steps typically *reduced* the brightness of these aluminum surfaces. That, in turn, detrimentally impacted the initial properties of aluminum products made thereby.
3. Many chemical treatment and painting processes were applied to enhance: (a) the adhesion of subsequent coatings to these aluminum products; and (b)
30 the corrosion resistance performance thereof. For any given product, a compromise had to be reached between greater brightness and greater durability.
4. From a manufacturing standpoint, past processes involved a large

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number of steps requiring relatively high levels of employee involvement to assure consistency and quality. That translates into high operating and production costs.

5 5. While maximum corrosion resistance may be achieved with hexavalent chromium, that component should be avoided because of its detrimental environmental and health risks.

Numerous processes for cleaning, etching, coating and/or surface treating aluminum products are known. They include: U.S. Patent Nos. 4,440,606, 4,601,796, 4,793,903, 5,290,424, 5,486,283, 5,538,600, 5,554,231, 5,587,209, 5,643,434 and 5,693,710.

10 In U.S. Patent No. 5,290,424, the disclosure of which is fully incorporated by reference herein, image clarity of a particular product, decorative reflective sheet made from 5000 or 6000 Series aluminum alloys, was improved. The present invention, by contrast, is not limited to just sheet product. It can also be used to surface treat aluminum extrusions, forgings and castings, especially those made
15 from Al-Mg alloys, Al-Mg-Si alloys, Al-Si-Mg alloys and/or copper-containing variants of the latter two alloys.

The present invention imparts brightness to the surface of aluminum products, especially vehicle wheels, while improving the adhesion, soil resistance and corrosion resistance performance of such products. This invention accomplishes the
20 foregoing property attributes through a manufacturing sequence that involves 25% fewer steps thereby reducing overall production costs. The invention combines two of the more costly known surface treatment steps, those of surface brightening and cleaning, into one step. At the same time, the method of this invention employs more user friendly components that pose no immediate or long term risks to operators
25 or the environment. Finally, because of the chemical nature of this process, resulting end products exhibit a higher abrasion resistance.

The new method of this invention consists of:

Main Step 1. A single chemical treatment, the composition and operating parameters of which are adjusted depending on whether the preferred
30 products to be treated are made from an Al-Mg, Al-Mg-Si or an Al-Si-Mg alloy. This chemical treatment step imparts brightness to the aluminum being treated while yielding a chemically clean outer surface ready for subsequent processing. This step

replaces previous multi-step buffing and chemical cleaning operations. On a preferred basis, this chemical brightening step uses an electrolyte with a nitric acid content between about 0.05 to 2.7 % by weight. It has been observed that beyond 2.7 wt % nitric acid, a desired level of brightness for Al-Mg-Si-Cu alloys cannot be achieved. On a preferred basis, the electrolyte for this step is phosphoric acid-based, alone or in combination with some sulfuric acid added thereto, and a balance of water.

Main Step 2. The second main step is to deoxidize the surface layer of said aluminum product by exposure to a bath containing nitric acid, preferably in a 1:1 dilution from concentrated. This necessary step "prep's" the surface for the oxide modification and siloxane coating steps that follow.

Main Step 3. The third main step of this invention is a surface oxide modification designed to induce porosity in the surface's outer oxide film layer. The chemical and physical properties resulting from this modification will have no detrimental effect on end product (or substrate) brightness. Like main step 1, the particulars of this oxide modification step can be chemically adjusted for Al-Mg-Si versus Al-Si-Mg alloys using an oxidizing environment induced by gas or liquid in conjunction with an electromotive potential. Surface chemistry and topography of this oxide film are critical to maintaining image clarity and adhesion of a subsequently applied polymeric coating. One preferred surface chemistry for this step consists of a mixture of aluminum oxide and aluminum phosphate with crosslinked pore depths ranging from about 0.01 to 0.1 micrometers, more preferably less than about 0.05 micrometers.

Main Step 4. Fourthly, an abrasion resistant, siloxane-based layer is applied to the aluminum product, said layer reacting with the underlying porous oxide film, from above step 3, to form a chemically and physically stable bond therewith. Preferably, this siloxane coating is sprayed onto the substrate using conventional techniques in which air content of the sprayed mixture is minimized (or kept close to zero). To optimize transfer onto the aluminum part, viscosity and volatility of this applied liquid coating may be adjusted with minor amounts of butanol being added thereto.

The foregoing method steps of this invention eliminate filiform

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corrosion while maintaining an initial brightness of the aluminum product to which they are applied. In some instances, the invention also imparts brightness to the product while yielding a chemically clean surface in fewer steps thereby reducing overall production costs. Finally, this invention imparts some degree of abrasion resistance, a major requirement for various aluminum products such as vehicle wheels made by forging, casting or other known or subsequently developed manufacturing practices. It accomplishes all of the foregoing without the use of environmentally risky or health threatening components.

Further features, objectives and advantages of this invention will be made clearer from the following detailed description of preferred embodiments made with reference to the accompanying drawings in which:

Figure 1 is a flowchart depicting the detailed main steps, and related substeps comprising one preferred treatment method according to this invention, said steps having occurred *after* the typical cleaning (alkaline and/or acidic) and rinse of aluminum products; and

Figures 2a and 2b are schematic, side view drawings depicting the aluminum alloy surfaces of a conventional clear coated product (Figure 2a) versus an enlarged side view layering from an aluminum product treated according to this invention (Figure 2b).

For any description of preferred alloy compositions and/or method treatment components herein, all references are to percentages by weight percent (wt.%) unless otherwise indicated. Also, when referring to any numerical range of values herein, such ranges are understood to include each and every number and/or fraction between the stated range minimum and maximum. A magnesium content range of about 0.8-1.2 wt %, for example, would expressly include all intermediate values of about 0.81, 0.82, 0.83 and 0.9%, all the way up to and including 1.17, 1.18 and 1.19% Mg. The same applies to every other elemental and/or operational range set forth below.

When referring to aluminum alloys throughout, terms such as 5000 and 6000 Series alloys, for example, are made with reference to Aluminum Association standards, the disclosures of which are fully incorporated by reference herein.

Prior to this invention, known practices for cleaning and coating a

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bright aluminum wheel product typically included the following individualized steps (or distinct activities): 1. A Multi-step Buff; 2. Clean; 3. Rinse; 4. Deoxidize; 5. Rinse; 6. Chemical Conversion; 7. Rinse; 8. Seal; 9. Rinse; 10. Oven Dry; 11. Powder Spray; and 12. Oven Cure. By contrast, the comparative stages of this invention, for the same wheel product, include: 1. Brightening; 2. Rinse; 3. Deoxidize; 4. Rinse; 5. Oxide Modification; 6. Rinse; 7. Dry; 8. Silicate; and 9. Cure. Through 25% fewer method steps, this invention manages to achieve better brightness, corrosion resistance and, for the first time, some enhanced abrasion resistance.

10 Method Step Particulars

Main step 1: Preferred chemical brightening conditions for this step are phosphoric acid-based with a specific gravity of at least about 1.65, when measured at 80°F. More preferably, specific gravities for this first main method step should range between about 1.69 and 1.73 at the aforesaid temperature. The nitric acid additive for such chemical brightening should be adjusted to minimize a dissolution of constituent and dispersoid phases on certain Al-Mg-Si-Cu alloy products, especially 6000 Series extrusions and forgings. Such nitric acid concentrations dictate the uniformity of localized chemical attacks between Mg₂Si and matrix phases on these 6000 Series Al alloys. As a result, end product brightness is positively affected in both the process electrolyte as well as during transfer from process electrolyte to the first rinsing substep. On a preferred basis, the nitric acid concentrations of main method step 1 should be about 2.7 wt.% or less, with more preferred additions of HNO₃ to that bath ranging between about 1.2 and 2.2 wt.%.

For optimum brightening, the surface treatment method of this invention should be practiced on 6000 Series aluminum alloys whose iron concentrations are kept below about 0.35% in order to avoid preferential dissolution of Al-Fe-Si constituent phases. More preferably, the Fe content of these alloys should be kept below about 0.15 wt % iron. At the aforementioned specific gravities, dissolved aluminum ion concentrations in these chemical brightening baths should not exceed about 35 g/liter. The copper ion concentrations therein should not exceed about 150 ppm.

Main step 2: A chemically brightened product is next subjected to

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purposeful deoxidation. One preferred deoxidizer suitable for wheel products made from 5000 or 6000 Series aluminum alloys is a nitric acid-based bath, though it is to be understood that still other known or subsequently developed deoxidizing compositions may be substituted therefor. For the nitric acid bath, a 1:1 dilution
5 from concentrate has worked satisfactorily.

After chemical brightening, remaining concentrations of Cu should be removed from the product surface to extend its overall durability. One means for accomplishing this is to adjust the nitric acid levels above so that Cu concentrations on the alloy surface does not exceed about 0.3 wt %.

10 Main step 3: Subsequent to deoxidation, an oxide modification step is performed that is intended to produce an aluminum phosphate and/or phosphonate film with the morphological and chemical characteristics necessary to accept bonding with a polymeric silicate coating. This oxide modification step should deposit a thickness coating of about 1000 angstroms or less, more preferably between about 75
15 and 200 angstroms thick. If applied electrochemically, this can be carried out in a bath containing about 2 to 15% by volume phosphoric or phosphonic acid.

Main step 4: The resultant properties of aluminum surfaces treated by to this invention are dependent on the uniformity, smoothness and adhesion strength of the final siloxane film layer deposited thereon. Siloxane-based chemistries are
20 applied to the oxide-modified layers from Step 3 above. Both initial and long term durability of such treated products depend on the proper surface activation of these metals, followed by a siloxane-based polymerization. Abrasion resistance of the resultant product is determined by the relative degree of crosslinking for the siloxane chemicals being used, i.e. the higher their crosslinking abilities, the lower the
25 resultant film flexibility will be. On the other hand, lower levels of siloxane crosslinking will increase the availability of functional groups to bond with modified, underlying Al surfaces thereby enhancing the initial adhesion strengths. Under the latter conditions, however, coating thicknesses will increase and abrasion resistance decreases leading to lower clarity and durability properties, respectively.

30 Overall, it is preferred that a hard siloxane chemistry be used with aluminum vehicle wheels made from 6000 Series alloys. Suitable siloxane compositions for use in main step 4 include those sold commercially by SDC

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Coatings Inc. under their Silvue® brand. Other suitable manufacturers of siloxane coatings include Ameron International Inc., and PPG Industries, Inc. It is preferred that such product polymerizations occur at ambient pressure for minimalizing the impact, if any, to metal surface microstructure.

5 For any given aluminum alloy composition and product form, the compatibility of main step 1 surface treatments with main step 4 siloxane polymerizations will dictate final performance attributes. Due to the stringent surface property requirements needed to achieve highly crosslinked siloxane chemical adhesion atop metal surfaces, highly controlled surface preparations and
10 polymerization under vacuum conditions are typically used. Most preferably, siloxane chemistries are applied using finely dispersed droplets rather than ionization in a vacuum. Control and dispersion of these droplets via an airless spray atomization minimizes exposure with air from conventional paint spraying methods and achieves a preferred breakdown of siloxane dispersions in the solvent. The end
15 result is a thin, highly transparent, "orange peel"-free coating.

Referring now to Figures 2a and 2b, there is shown two side view schematics comparing the deposits of a conventional prior art, clear coat process (Figure 2a) versus the surface treatment layers deposited according to this invention (Figure 2b). For vehicle wheels, the most widely used system for conversion coating
20 is to apply powder coats using conventional acrylic or polyester chemistries. Such paint chemistries provide accessible functional groups for adhesion to the metal surface, but their adhesion strengths and durabilities are dependent on the interfacial properties of the metal alloy/conversion coat/paint system employed.

For the present invention, a diffuse interface has been postulated which
25 minimizes the probability of coating delamination from the treated metal surface. This is achieved by replicating highly controlled surface modification processes to yield an aluminum phosphate or phosphonate with the proper microstructure and morphology such that siloxane chemistry adhesions are accomplished at ambient pressure. The preferred siloxane based chemicals described above also result in a coating thickness
30 approximately one order of magnitude smaller than those deposited using acrylic or polyester powders. It is believed that these carefully selected and preferably customized chemistries result in a coating with higher uniformity and transparency

(i.e. clarity) than was possible before. In terms of hydrophobicity and permeability, siloxane based chemistries also yield more water repellent properties and lower water permeability than their acrylic and polyester coating counterparts. This results in an easier to clean, durable aluminum coated surface, in various product forms.

5

Experimental Results:

Using three different standards of corrosion performance, those established by General Motors, Ford, and ASTM Standard G85, the particulars of which are all fully incorporated by reference, aluminum wheel products treated according to this invention fared favorably well compared to a second wheel (same alloy composition) treated per the known, prior art 12-step process described above.

10

Process	GM 9682P	FORD FLTM B1 124-01	ASTM G85
12 Step	2.0-2.5 mm	2.0-3.0 mm	3.0 mm (2 wks)
Invention	0 mm	0 mm	0 mm

15

Heavy duty vehicle wheels experimentally treated by the method of this invention were subjected to standard road conditions through several seasons, and to coarser, off-road, construction type conditions. In both cases, these wheels were periodically cleaned (approximately monthly) using pressurized water sprays, with and without soaps, to reveal, repeatedly, the shiny, transparent and still dirt resisting aluminum surfaces underneath.

20

Having described the presently preferred embodiments, it is to be understood that the invention may be otherwise embodied by the scope of the appended claims.

C L A I M S

1. A method for surface treating an aluminum product to improve its brightness, said method comprising the main steps of:
 - (a) applying a chemical brightening composition to the product;
 - 5 (b) deoxidizing the product surface in a nitric acid-based bath;
 - (c) forming a porous oxide on said product surface by contacting with an electrolytic bath containing phosphoric or phosphonic acid; and
 - (d) applying a siloxane-based outer layer to the porous oxide.
2. The method of claim 1, wherein said aluminum product is made from
10 a 5000 or 6000 Series aluminum alloy (Aluminum Association designation).
3. The method of claim 2, wherein said aluminum alloy is a 5000 Series alloy selected from the group consisting of: 5454, 5182 and 5052 aluminum.
4. The method of claim 2, wherein said aluminum alloy is a 6000 Series alloy selected from the group consisting of: 6061, 6063 and 6005 aluminum.
- 15 5. The method of claim 4, wherein said alloy contains less than about 0.35 wt.% iron.
6. The method of claim 5, wherein said alloy contains less than about 0.15 wt.% iron.
7. The method of claim 1, wherein said aluminum product is selected
20 from the group consisting of an extrusion, a forging and a casting.
8. The method of claim 1, wherein said aluminum product is a vehicle wheel.
9. The method of claim 1, wherein said aluminum product is subjected to cleaning and rinsing prior to step (a).
- 25 10. The method of claim 9, wherein said pre-step (a) cleaning is alkaline-based.
11. The method of claim 9, wherein said pre-step (a) cleaning is acid-based.
12. The method of claim 1, wherein said aluminum product is subjected to
30 a rinsing substep after one or more of steps (a), (b) or (c).
13. The method of claim 1, wherein the chemical brightening composition of step (a) includes: about 2.7 wt % or less nitric acid, about 70-90 wt %

phosphoric acid, the balance water and impurities.

14. The method of claim 13, wherein said chemical brightening composition contains about 1.2-2.2 wt % nitric acid.
15. The method of claim 1, wherein said oxide forming step (c) includes
5 contacting the product with an electrolytic bath containing about 2 to 15 vol % phosphoric or phosphonic acid.
16. The method of claim 15, wherein the oxide that is formed is about 1000 angstroms thick or less.
17. The method of claim 16, wherein said oxide is about 75 and 200
10 angstroms thick.
18. The method of claim 1, wherein said siloxane-based film is applied by spray coating.
19. The method of claim 1, wherein said aluminum product is subjected to air drying after step (d).
- 15 20. The method of claim 19, wherein said siloxane-based film is thermally cured after air drying.
21. A method for surface treating aluminum wheel products to improve their brightness and abrasion resistance, said method comprising the steps of:
- 20 (a) applying a chemical brightening composition to said wheel products;
- (b) deoxidizing the surface of said wheel products in a nitric acid-based bath;
- (c) forming a porous oxide on said surface by contacting with an electrolytic bath containing phosphoric or phosphonic acid;
- 25 (d) applying a siloxane-based film to the porous oxide; and
- (e) thermally curing the siloxane-based film on said surface.
22. The method of claim 21, wherein said wheel products are made from a 5000 or 6000 Series aluminum alloy (Aluminum Association designation).
23. The method of claim 22, wherein said aluminum alloy is a 5000 Series
30 alloy selected from the group consisting of: 5454, 5182 and 5052 aluminum.
24. The method of claim 22, wherein said aluminum alloy is a 6000 Series alloy selected from the group consisting of: 6061, 6063 and 6005 aluminum.

25. The method of claim 22, wherein said aluminum alloy contains less than about 0.35 wt.% iron.
26. The method of claim 25, wherein said aluminum alloy contains less than about 0.15 wt.% iron.
- 5 27. The method of claim 21, wherein said wheel products are subjected to cleaning and rinsing prior to step (a).
28. The method of claim 27, wherein said pre-step (a) cleaning is alkaline-based.
29. The method of claim 27, wherein said pre-step (a) cleaning is acid-
10 based.
30. The method of claim 21, wherein said wheel products are subjected to a rinsing substep after one or more of steps (a), (b) or (c).
31. The method of claim 21, wherein the chemical brightening composition of step (a) includes: about 2.7 wt % or less nitric acid, about 70-90 wt %
15 phosphonic acid, the balance water and impurities.
32. The method of claim 31, wherein said chemical brightening composition contains about 1.2-2.2 wt % nitric acid.
33. The method of claim 23, wherein said oxide forming step (c) includes contacting said wheel products with an electrolyte containing about 2 to 15 vol %
20 phosphoric or phosphonic acid.
34. The method of claim 33, wherein the oxide that is formed is about 1000 angstroms thick or less.
35. The method of claim 34, wherein said oxide is about 75 and 200 angstroms thick.
- 25 36. The method of claim 21, wherein said siloxane-based film is applied by spray coating.
37. A method for surface treating cleaned and rinsed, 6000 Series aluminum wheel products to improve their brightness, soil and abrasion resistance, said method comprising the steps of:
- 30 (a) chemically brightening said wheel products with a composition that includes phosphoric acid and nitric acid;
- (b) rinsing said wheel products;

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(c) deoxidizing the surface of said wheel products in a nitric acid-based bath;

(d) rinsing said wheel products;

(e) forming a porous oxide on said surface by contacting with an electrolytic bath containing phosphoric or phosphonic acid;

(f) rinsing said wheel products;

(g) applying a siloxane-based film to said oxide; and

(h) thermally curing the siloxane-based film on said wheel products.

38. The method of claim 37, wherein said 6000 Series aluminum is selected from the group consisting of: 6061, 6063 and 6005 alloys.

39. The method of claim 38, wherein said alloy contains less than about 0.35 wt.% iron.

40. The method of claim 39, wherein said alloy contains less than about 0.15 wt.% iron.

41. The method of claim 38, wherein said wheel products are cleaned and rinsed prior to step (a).

42. The method of claim 41, wherein said pre-step (a) cleaning is alkaline-based.

43. The method of claim 41, wherein said pre-step (a) cleaning is acid-based.

44. The method of claim 37, wherein the composition of step (a) includes: about 2.7 wt % or less nitric acid, about 70-90 wt % phosphonic acid, the balance water and impurities.

45. The method of claim 44, wherein said chemical brightening composition contains about 1.2-2.2 wt % nitric acid.

46. The method of claim 38, wherein the oxide forming step (c) includes contacting said wheel products with an electrolytic bath containing about 2 to 15 vol % phosphoric or phosphonic acid.

47. The method of claim 46, wherein the oxide that is formed is about 1000 angstroms thick or less.

48. The method of claim 47, wherein said oxide is about 75 and 200 angstroms thick.

49. The method of claim 37, wherein said aluminum product is air dried after step (g).

FIG. 1

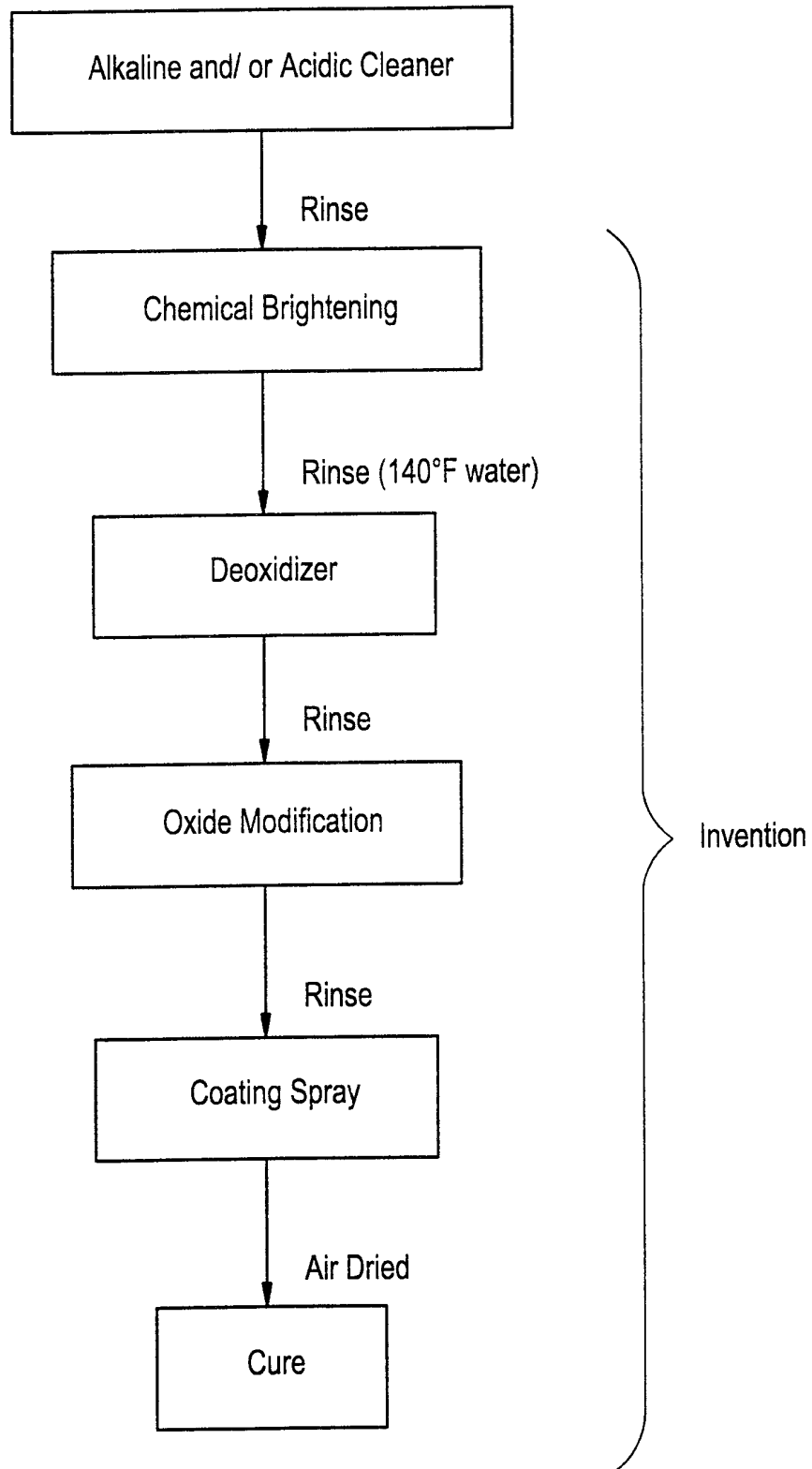


FIG. 2A
PRIOR ART

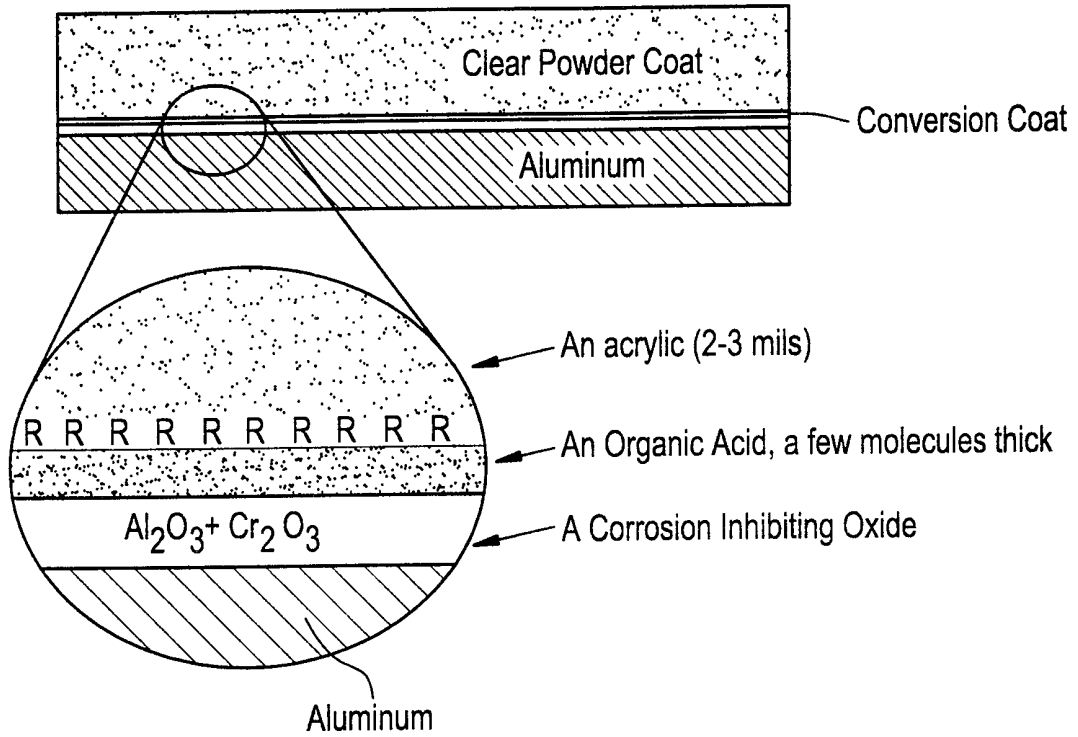


FIG. 2B

